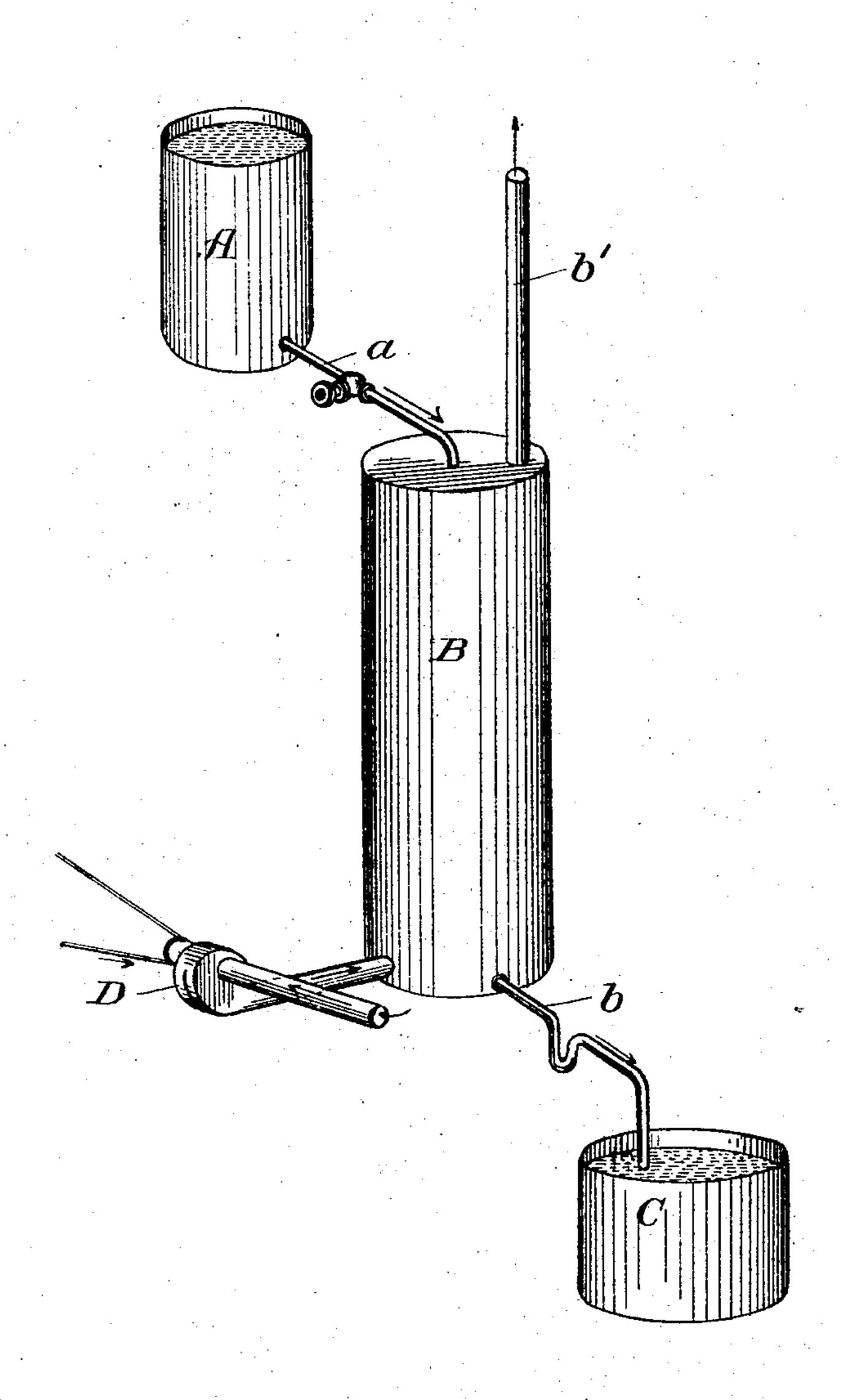
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H. H. DOW.

METHOD OF CONVERTING BROMIN INTO BROMIDS AND BROMATES.

APPLICATION FILED FEB. 13, 1902. RENEWED DEC. 29, 1903.

NO MODEL.



Witnesses,

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METHOD OF CONVERTING BROMIN INTO BROMIDS AND BROMATES.

SPECIFICATION forming part of Letters Patent No. 765,417, dated July 19, 1904.

Application filed February 13, 1902. Renewed December 29, 1903. Serial No. 187,070. (No specimens.)

To all whom it may concern.

Be it known that I, HERBERT H. Dow, a citizen of the United States, and a resident of Midland, county of Midland, and State of Michigan, have invented a new and useful Improvement in Methods of Converting Bromin into Bromids and Bromates, of which the following is a specification, the principle of the invention being herein explained and the best mode in which I have contemplated applying that principle, so as to distinguish it from other inventions.

My invention relates to methods of converting free bromin into bromids and bromates or its equivalent hypobromite, its object being to effect such conversion in a manner more economical than has heretofore been the practice.

Said invention consists of a process hereinafter fully described, and specifically set forth in the claims.

Heretofore alkaline bromids have been formed from free bromin by combining the latter with a hydrate of the alkali in solution; but if the source from which the bromin is being obtained contains any carbon dioxid the caustic solution will be converted first into a carbonate and then into a bicarbonate of the alkali. The bromin alone or in solution without excess of air will not combine with a bicarbonate of an alkali, but will slowly combine with the normal carbonate as shown by the following equation:

 $_{35}^{6}$ $_{6}^{6}$ $_{6}^{6}$ $_{1}^{6}$ $_{1}^{6}$ $_{1}^{6}$ $_{2}^{6}$ $_{2}^{6}$ $_{3}^{6}$ $_{1}^{6}$ $_{1}^{6}$ $_{2}^{6}$ $_{3}^{6}$ $_{1}^{6}$ $_{3}^{6}$ $_{4}^{6}$ $_{5}^{6}$

the bromin hence combining with but one-half the potassium present.

The annexed drawing and the following description set forth in detail one mode of carrying out the invention, such disclosed mode constituting but one of various ways in which the principle of the invention may be used.

Said annexed drawing illustrates diagrammatically an apparatus which is used for carrying out my improved process.

A receptacle A is provided with a suitable connection a, by means of which its contents may be discharged into the top of a coke,

lunge-plate, or other similar tower B, by 50 means of which a liquid may upon passage therethrough be comminuted, as will be understood, in order to give it a large superficial area. The bottom of the tower is provided with a connection b, which discharges into a 55 suitable receptacle C. A blower D is connected with the bottom of the tower and forces bromin-laden air up through same, such air being discharged into the atmosphere through a suitable vent b'. The bromin-laden air is 60 obtained by means of any known process, such as that described in my reissued Letters Patent No. 11,232, dated April 12, 1892.

My method consists first in bringing bromin-laden air into contact with the carbonates 65 or bicarbonates of any of the alkaline metals either in solution or in a solid state, such bromin-laden air being such as has not been previously used for the same purpose and being hence practically free from carbon dioxid. 70 By air practically free from carbon dioxid I mean atmospheric air to which no carbon dioxid has been artificially added—that is, atmospheric air containing a normal amount of carbon dioxid. The amount of carbon di- 75 oxid, I have found, should not exceed one per cent. by volume, and the smaller the percentage the more complete the conversion. Such air is after having been subjected to the action of such carbonates or bicarbonates and 80 so freed from bromin and containing the carbon dioxid liberated in the reaction discharged into the atmosphere, thus carrying off and removing completely from the system the carbon dioxid set free in the reaction, as 85 is hereinafter more fully explained.

One method of carrying out such process by means of the apparatus above described is to bring a cold solution of potassium carbonate, (K₂CO₃,) placed in receptacle A, into contact with the bromin-laden air forced through the tower B by blower D, the reactions being apparently as follows:

 $6K_2CO_3 + 6Br + 3H_2O =$

 $5KBr+KBrO_3+6KHCO_3$ 95

and $6KHCO_3+6Br+air=$ $5KBr+KBrO_3+3H_2O+6CO_2+air.$

When bromin unaccompanied by a large excess of air practically free from carbon dioxid is brought into contact with a solution of potassium carbonate, only the first reaction 5 above named takes place, and bromin under these conditions will not as previously stated, combine with the potassium bicarbonate formed, so that only one-half of the potassium in the normal carbonate is available for 10 the production of bromid and bromate; but I have found that when the bromin is accompanied by a large excess of air practically free from carbon dioxid nearly all of the alkali metal present may be made to combine with 15 the bromin, carbon dioxid being set free and carried off by the large excess of air present.

In the method as described in my said reissued Letters Patent the air used as a carrier of the bromin from the brine to the absorbent was used over and over again. By this means it is not possible to convert more than about one-half the alkaline metal in a normal carbonate into bromid and bromate, because the carbon dioxid at first set free from the bicarbonate formed is not removed, but remains in the system and continues to circulate in contact with the absorbing solution. Thus a condition of equilibrium is soon established, when no further amount of bicarbonate will be decomposed.

In my new method the bromin-laden air after passing over a very large absorbing-surface, by means of which nearly all the bromin is absorbed, is then discharged directly into the atmosphere, thus completely removing from the system the carbon dioxid set free. By this means I am able on a commercial scale to convert from ninety to ninety-seven per cent. of the alkali metal present into bromid and bromate.

Instead of air any other gas inert as regards the chemical action upon the particular substances used in the process and practically free from carbon dioxid may be used, and as an absorbent material the carbonate of any metal may be employed.

In the above reactions a certain amount of hypobromite is formed in place of part of the bromid and bromate; but the substance is quite on the substance is quite or stable and is ultimately converted into bromid and bromate.

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Other modes of applying the principle of my invention may be employed instead of the one explained, change being made as regards the process herein disclosed provided the 55 means stated by any one of the following claims or the equivalent of such stated means be employed.

A free access of air furnished by any suitable means, such as disclosed herewith, will 60 furnish the excess required to effect the ready and complete reaction of bromin with an alkaline bicarbonate if fresh air is continually supplied, and the air which has once been used and contains the carbon dioxid set free from 65 the bicarbonate is discharged from the apparatus. It will be noted that the principle of my invention is effected either when bromine is brought into contact with a carbonate or a bicarbonate, in the former case the bicarbonate 7° being first formed and then the bromid and bromate if a continuous supply of fresh air is furnished to the apparatus, the process thus taking place in two reactions, as has been described above.

I therefore particularly point out and distinctly claim as my invention—

1. In a method of converting bromin into bromid and bromate the steps which consist, in bringing bromin and a fresh gas practically 80 free from carbon dioxid into contact with an alkaline bicarbonate.

2. In a method of converting bromin into bromid and bromate the steps which consist, in bringing bromin and fresh gas practically 85 free from carbon dioxid simultaneously into contact with an alkaline bicarbonate.

3. In a method of converting bromin into bromid and bromate the steps which consist, in bringing bromin in combination with fresh 9° air practically free from carbon dioxid into contact with an alkaline bicarbonate.

4. In a method of converting bromin into bromid and bromate the steps which consist, in bringing bromin in combination with fresh 95 air practically free from carbon dioxid into contact with potassium bicarbonate.

Signed by me this 7th day of February, 1902. HERBERT H. DOW.

Attest:

A. E. MERKEL, D. T. DAVIES.